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Docket No.: KCC-17,458

THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Roger Bradshaw QUINCY, III

Group No.: 3761

Serial No: 10/037,466

Filing Date: 21 December 2001

Examiner:
Catharine L. Anderson

Title: ANTIMICROBIAL NONWOVEN WEBS FOR
PERSONAL CARE ABSORBENT ARTICLES

**DECLARATION OF ROGER B. QUINCY, III
PURSUANT TO 37 C.F.R. §1.131(a)**

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

I, Roger B. Quincy, III, declare as follows:

1. I am employed by the Kimberly-Clark Corporation. I am the sole inventor of the subject matter claimed in U.S. Patent Application Serial No. 10/037,466, entitled "ANTIMICROBIAL NONWOVEN WEBS FOR PERSONAL CARE ABSORBENT ARTICLES."

2. Prior to 06 September 2001, I conceived the idea of treating a fibrous nonwoven web with a halogenated polystyrene hydantoin in which only the amide nitrogen atoms, and not the imide nitrogen atoms, would be halogenated. Halogenated polystyrene hydantoins having chlorine atoms linked to both (amide and imide) nitrogen atoms had been shown to exhibit biocidal activity, but released unacceptable levels of free chlorine. I proposed that only partial chlorination of the polystyrene hydantoin, to fill the amide site but not the imide site, could produce a material that is more stable yet less active.

I hereby certify that this correspondence (along with any paper referred to as being attached or enclosed) is being deposited with the United States Postal Service as First Class Mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450 on

02 June 2004

02 June 2004

Date

Marky Retum

Signature

3. Prior to 06 September 2001, I approached HaloSource Corporation ("HaloSource") with my proposal. HaloSource had previously supplied a chlorinated polystyrene hydantoin designated Poly-1-C1, believed to have chlorine linkages present on imide and amide nitrogen sites. Following my proposal, HaloSource provided me with a new version of chlorinated polystyrene hydantoin, designated Poly-1-C1, Type 2, responsive to my proposal.

4. Prior to 06 September 2001, I treated nonwoven fabric samples with Poly-1-C1, Type 2, and evaluated the samples for biocidal activity. The treated fabric samples exhibited useful levels of biocidal activity and substantially reduced levels of free chlorine emission. This work has been documented on the following pages of my laboratory notebooks, copies of which are enclosed with dates redacted:

Laboratory Notebook P-7259, page 119; and

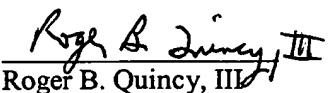
Laboratory Notebook P-7952, pages 59-62 and 69-80.

All of the foregoing notebook entries were documented prior to 06 September 2001.

5. All statements herein based on my own knowledge are true, and all statements made on information and belief are believed to be true. I acknowledge that willful false statements and the like are punishable by fine or imprisonment, or both (18 U.S.C. §1001) and may jeopardize the validity of this patent application or any patent issuing thereon.

6. Because of my unfamiliarity with the preparation of legal documents, I have been assisted in drafting this Declaration by an attorney of record, Maxwell J. Petersen.

Respectfully submitted,



Roger B. Quincy, III

Date: May 21, 2004



Corporation Serial No... P-7259
Lab. or Department... Non-work Technology
Project No. or Nos.
Date of First Entry
Date of Last Entry
Lab. or Dept. Head
Investigator ... Roger A. Quinney
Investigator
Investigator

LABORATORY NOTEBOOK

Treatment for Odor Control

Charged to Roger A. Quinney Date
Signatures of Investigators

.....
.....
.....
.....

Approved By
UNIT MANAGER
DEPARTMENT
DATE
.....

TITLE N-Halomic Chlorine Binding Idea

PROJECT NO. 57424

BOOK NO. P-7259

From Page No. —

At a meeting with Halosome at Neenah Conference City on [REDACTED], we were discussing the 2 chlorine binding sites on the hydantoin moiety attached to polythylene (called polythylene hydantoin or PSH). One of two chlorine is more labile than the other.

After the PSH has been converted to Poly-1-Cl (polythylene N-Halomic), I asked Halosome (Jeff Williams, et al.) if we could get a Poly-1-Cl that had only the tightly bound chlorine, thus figure out a way to provide a structure with only tightly bound chlorine (no labile chlorine since this would be a product poly concern).

RB Dings [REDACTED]

Quincy, Roger

From: Quincy, Roger
 Sent: Wednesday, [REDACTED] 5:34 PM
 To: Gadsby, Elizabeth
 Subject: RE: Invention Disclosure

Thanks for the feedback. I'll think about it. I'm more interested in trying to see if the PSH can be attached to SAP and become active via a moist bleach environment. Maybe it would be best for now to document the suggestion in your minutes so we have a record of proposing the idea to HaloSource. I would expect they could use a hydantoin that has only 1 hydrogen available for conversion to Cl or Br, instead of the 2 hydrogens on their PSH. They should also be able to put electron withdrawing groups on the hydantoin adjacent to the H which might bind the chlorine more tightly. I'll have to discuss this more with Y. Liu, our resident organic chemist.

—Original Message—
 From: Gadsby, Elizabeth
 Sent: Tuesday, [REDACTED] 4:03 PM
 To: Quincy, Roger
 Subject: Invention Disclosure

Roger:

As I am preparing the meeting minutes from the HaloSource meeting, I remembered the excellent idea that you had about manipulating the strength of the chlorine-binding sites. I was wondering if you wanted to consider preparing an invention disclosure on the concept. I think it is unique and HaloSource expressed that they had not been considering the needs to strengthen the bonds. It is also, a possible modification we would want to use to allow us to have improved product aging, processability, and maybe safety.

Best regards,
 Elizabeth

To Page No.

Work done by Roger B Dings

Date [REDACTED]

Witnessed

Recorded by Roger B Dings

Date [REDACTED]

Witnessed



Corporation Serial No. P-7952
Lab. or Department NT
Project No. or Nos. 57596
Date of First Entry
Date of Last Entry
Lab. or Dept. Head
Investigator Roy B. Dury
Investigator
Investigator

LABORATORY NOTEBOOK

Odor control strategies

Charged to Roy B. Dury Date
Signatures of Investigators
.....
.....
.....
.....

Approved By
UNIT MANAGER
DEPARTMENT
DATE
.....

FILE Poly-Cl Type-2

From Page No. — Background: At a meeting with Helselme Corp. at Neenah Conference Center on [REDACTED], I requested that Helselme provide K-C with a Poly-Cl (polytype N-Helme) that contained only tightly bound chlorine (see formulation of the idea on p. 119, P-7959). Helselme claims to have been able to reduce the idea to practice and has supplied Poly-Cl Type-2 for our evaluation.

Proposed Evaluation for Poly-Cl Type-2:

- ① Test the headspace above the particles with a Chlorine Drieger tube. This will tell us if any volatile chlorine compounds are being given off by the Poly-Cl Type-2 particles.
- ② Test a fiber structure that contains Poly-Cl Type-2 particles with the Chlorine Drieger tube to see if any volatile chlorine compounds are being given off.
- ③ Evaluate antimicrobial properties for a fiber structure that contains Poly-Cl Type-2 particles.

Prep of fiber structure with Poly-Cl Type-2:

— using the Neenahs Handiset Form (HSF) to produce 500 gsm. sheet with 5% Poly-Cl Type-2 (lot H5039-77-3, rec. for Helme 7/01) and without any Poly-Cl Type-2.

The handiset form area is 0.258 m². A piece of tissue lining paper was placed on top of the plate (~0.5 cm each side) in the HSF box. The filtered pulp (believed to be SW16) was divided into 4 sections (122.5 g total weight before dividing). Poly-Cl Type-2 (6.75 g) was distributed among the 4 pulp sections. The Poly-Cl Type-2 + pulp was folded like a towel and pieces were broken off and fed into the HSF head. The conditions used for the HSF were: dust collector vacuum on, vacuum pump on top setting (60 Hz), 40 psi, pulsed air into HSF, 4000 rpm blade speed. (cont)

To Page No. 60

Work done by
Roger B. Loring, R.E.
Reviewed byDate [REDACTED]
0-10 [REDACTED]Witnessed
[REDACTED]

Date [REDACTED]

NAME Poly-L-L Type-2

From Page No. 59 Contd.

PROJECT NO. 57596
BOOK NO. P-7952

BOOK NO. P-7952

A control (500 gsm fluffy sheet without Poly-Cl Type-2) was made with the same HGF conditions (see p. 59, P-7952). 1.9 g of the fibrillized pulp was used.

The two backsheets were deformed by applying a light coat of 166-1075 (mixed with (100)) to both sides (each backsheet had a tissue layer surrounding the poly layer) using a spray apparatus (description can be seen on p. 193, P-6958), followed by pressing with a Carver Press (no heat) to a thickness of 0.125 inch (4 0.125 inch plies are placed between the two plates) for 5 minutes. Each of the rigid plates was cut into four

Also, a piece of release paper was placed on the top and bottom of each $9'' \times 9''$ section before pressing with the Corval press. This kept the top and bottom of each section from coming in contact with the steel plates of the press. The 4 sections from each of the two backsets were layered out on a surface (e.g. chair) to dry overnight. The backsets and sections will be labeled as follows:

label

Description

7952-60-PA

5'2. Poly-L-L Type-2 / poly L-Aspartic section A

7452-60-PB

~~7952-60-PC~~

8 5 4 3 2 1

3952-60-PD

u u u u u u u

To Page No. 61

Work done by Ron B. Jones, REN

Date _____

Wincsod

Recorded by

Boyle & Son

152

Whales

Date

Proprietary, To Be Maintained in Confidence

PROPERTY OF KIMBERLY-CLARK CORPORATION

61

TITLE Poly 1-6 Type-2

PROJECT NO. 57596
BOOK NO. P-7952

From Page No. 60

Cont.

<u>Label</u>	<u>Description</u>									
7952-61-CA	poly 1-6 Type-2 Control portion A									
7952-61-CB	n n n n B									
7952-61-CC	n n n n C									
7952-61-CD	n n n n D									
<hr/>										
<u>Antimicrobial Evaluation</u>										
A $\frac{4}{5} \times \frac{4}{5}$ piece of the following codes will be sent to Viramed Biologics Laboratories $\frac{5}{5} \times \frac{5}{5}$ (soon to be AppTec Laboratories Inc) for ATCC method 100 (modified). Three microorganisms will be tested (S. aureus ATCC 6538, E. coli ATCC 8739, P. mirabilis ATCC 4630) and 5 dilutions will be done (down to 10^1 Colony forming units (CFU)).										
<u>Codes for Viramed</u>										
7952-60-PA	- PB - PC - PD									
7952-61-CB	- CD									

To Page No. 62

Work done by <i>Roy B. Zing</i>	Date <i>[Redacted]</i>	Witnessed <i>[Signature]</i>	Date <i>[Redacted]</i>
Recorded by <i>Roy B. Zing</i>	Date <i>[Redacted]</i>	Witnessed <i>[Signature]</i>	Date <i>[Redacted]</i>

PROPERTY OF KIMBERLY-CLARK CORPORATION

TITLE Poly 1-Cl Type-2

57596
P-7952

From Page 11 61

ViroMed letter:



Kimberly-Clark

RQ

Ms. Karen Alexander
ViroMed Biosafety Laboratories
1265-B KenneStone Circle
Marietta, GA 30066

Dear Ms. Alexander,

Please find six fabric samples (5" by 5") for microbiology test AATCC Method 100 (modified). As we discussed earlier this week, I would like to have 3 organisms (S. aureus ATCC 6538, E. coli ATCC 8739, P. mirabilis ATCC 4630) tested per sample using a 4 hour contact time with 5 dilutions. For reference, a similar procedure (only 4 dilutions) can be found in a previous report sent to me (Report Number: F0816018). The purchase order for this work is #15VB8653CB. Thanks for your help. Please contact me if you have any questions.

Sincerely,

Roger Quincy
Roger Quincy
Nonwoven Technology



Kimberly-Clark

1400 Holcomb Bridge Road
Roswell, GA 30076-2199
(770) 587-7884
(770) 587-7703 Facsimile
E-mail: rquincy@kcc.com

Roger B. Quincy, Ph.D.
Nonwoven Technology

Kimberly-Clark Corporation

1400 Holcomb Bridge Road Roswell, Georgia 30076 (770) 587-8000

Work done by

Roger B. Quincy

Recorded by

Roger B. Quincy

OE Dafin

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Print above To be Determined. Questions on

PROPERTY OF KIMBERLY-CLARK CORPORATION

PROJECT NO. 57596
SICK NO. P-7952TITLE Poly 1 - Cl Type - 262 Results from ViroMedR&Q

12:21 VIROMED → 7705877703

NO.517 D02



ViroMed Laboratories, Inc.

CLIENT TECHNICAL PROCEDURE

UNCONTROLLED COPY

Document #: GM210KMC.01

QA Approval: J. Thompson

Section: General Microbiology

Technical Approval: J. CalavenderEffective Date:

Revision #: 1

Page 1 of 1

Client: Kimberly ClarkProduct(s): Absorbent FabricTest Method: AATCC Test Method 100 (Modified)

Procedure:

Test Portion: 4-5 cm squareContainer: Petri DishTest Organism: (1) S. aureus ATCC # 6538, (2) E. coli ATCC # 8739, (3) P. mirabilis ATCC # 4630Inoculum level: Approximately 10⁶Time Intervals/
Incubation: 0° / NA, 4 hours / 35°C - 39°CDiluent: Lethen BrothAmount: 100 mLExtraction Method/Time: Stomaching / 2 minutes

Plating and Incubation:

Aliquot or
Dilution Plated: 10⁻¹, 10⁻², 10⁻³, 10⁻⁴, 10⁻⁵Culture Medium: Tryptic Soy AgarTemperature: 30°-35°CTime: 2 daysComments: NAForm # GMCTP.1
Rev. 1

CONFIDENTIAL

1265-B Kennesaw Circle • Marietta, GA 30068 • 888.847.6633 • 770.514.0262 • Fax 770.514.0294

70Work done by ViroMedDate Witnessed Recorded by Rog A. DuryDate Witnessed

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PROPERTY OF KIMBERLY-CLARK CORP/CLARK

57596
P-7952

TITLE: Poly-Cl Type-2

From Page No. 69

VIROMed Laboratories, Inc.

ViroMed's biosafety laboratories
are becomingAppTec
LABORATORY

The ViroMed biosafety laboratories are becoming AppTec Laboratories. AppTec is a leading provider of environmental monitoring services. Please direct any inquiry to the AppTec laboratory.

Report Number:
G0823032Kimberly-Clark
1400 Holcomb Bridge Road
Roswell, GA 30076-2199

P.O. #: 15VB8653CB

Attn: Dr. Roger B. Quincy

GENERAL MICROBIOLOGY TEST REPORT

Sample Information: Fabric Sample, 7952-61-CD

Date Received:

Date in Test:

Date Completed:

Test Information:

AATCC Method 100 (modified)

Procedure #: GM210KMC.01

Culture Medium: Tryptic Soy Agar

Neutralizer Solution: Lethen Broth

Test Sample and Organism	<i>S. aureus</i> ATCC 6538	<i>E. coli</i> ATCC 8739	<i>P. mirabilis</i> ATCC 4630
Inoculum Concentration	8.4×10^6	4.7×10^7	3.4×10^6
Initial Contact Time	1.1×10^7	4.5×10^7	3.7×10^6
4 Hour Contact Time	1.7×10^7	5.1×10^7	6.2×10^5
Percent Reduction	NR	NR	83.24%

NR = No Reduction

Katia Bartolotti

Departmental Review

Karen Alexander

Technical Review

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Work done by
ViroMedRecorded by
Roger B. Quincy

Printed Name: Roger B. Quincy

Joe J. SaylorAT

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Solv-C Type-2

PROJECT NO. 57596
STOCK NO. P-795270 Cont.

RBQ

ViroMed Laboratories, Inc.

ViroMed's biosafety laboratories
are becomingReport Number:
G0823029Kimberly-Clark
1400 Holcomb Bridge Road
Roswell, GA 30076-2199

P.O. #: 15VB8653CB

Attn: Dr. Roger B. Quincy

GENERAL MICROBIOLOGY TEST REPORT

Sample Information: Fabric Sample, 7952-61-CB

Date Received: [REDACTED]Date in Test: [REDACTED]Date Completed: [REDACTED]Test Information: AATCC Method 100 (modified)
Procedure #: GM210KMC.01
Culture Medium: Tryptic Soy Agar
Neutralizer Solution: Lethen Broth

Test Sample and Organism	<i>S. aureus</i> ATCC 6538	<i>E. coli</i> ATCC 8739	<i>P. mirabilis</i> ATCC 4630
Inoculum Concentration	8.4×10^6	4.7×10^7	3.4×10^6
Initial Contact Time	5.1×10^6	2.8×10^7	2.3×10^6
4 Hour Contact Time	1.3×10^7	2.9×10^7	3.1×10^5
Percent Reduction	NR	NR	86.52%

NR = No Reduction

Karen Bartlett
 Departmental Review

Karen Alexander
 Technical Review

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AEG

72

 ViroMed
 Roger B. Quincy

Witnessed		Date
Witnessed		Date
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Poly-CU Type-2

57596
P-7952

71 Contd

R&Q



ViroMed

Laboratories, Inc.

ViroMed's biosafety laboratories
are becoming



For additional information, call 800.345.3000
or visit our website at www.viromed.com
for a copy of our Quality System Manual.

Report Number:
G0823027

Kimberly-Clark
1400 Holcomb Bridge Road
Roswell, GA 30076-2199

P.O. #: 15VB8653CB

Attn: Dr. Roger B. Quincy

GENERAL MICROBIOLOGY TEST REPORT

Sample Information: Fabric Sample, 7952-60-PD

Date Received:

Date in Test:

Date Completed:

Test Information:

AATCC Method 100 (modified)

Procedure #: GM210KMC.01

Culture Medium: Tryptic Soy Agar

Neutralizer Solution: Lethen Broth

Test Sample and Organism	<i>S. aureus</i> ATCC 6538	<i>E. coli</i> ATCC 8739	<i>P. mirabilis</i> ATCC 4630
Inoculum Concentration	8.4×10^6	4.7×10^7	3.4×10^6
Initial Contact Time	2.7×10^7	8.7×10^8	1.9×10^5
4 Hour Contact Time	3.0×10^1	1.0×10^1	5.5×10^1
Percent Reduction	99.99%	99.99%	99.97%

Katherine Forrester

Departmental Review

Karen Alexander

Technical Review

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Key

73

ViroMed
Roger B. Quincy

RG/MB

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TITLE *Poly 1-Cl Type-2*PROJECT NO. 57596
BOOK NO. P-79272 *Cont.**ABQ*

ViroMed Laboratories, Inc.

ViroMed's biosafety laboratories
are becomingReport Number:
G0823030Kimberly-Clark
1400 Holcomb Bridge Road
Roswell, GA 30076-2199P.O. #: 15VB8653CB

Attn: Dr. Roger B. Quincy

GENERAL MICROBIOLOGY TEST REPORT

Sample Information: Fabric Sample, 7952-60-PC

Date Received:

Date in Test:

Date Completed:

Test Information:

AATCC Method 100 (modified)
Procedure #: GM210KMC.01
Culture Medium: Tryptic Soy Agar
Neutralizer Solution: Lethen Broth

Test Sample and Organism	<i>S. aureus</i> ATCC 6538	<i>E. coli</i> ATCC 8739	<i>P. mirabilis</i> ATCC 4630
Inoculum Concentration	8.4×10^6	4.7×10^7	3.4×10^8
Initial Contact Time	8.7×10^6	2.3×10^6	See Note
4 Hour Contact Time	$< 1.0 \times 10^1$	1.2×10^2	1.0×10^1
Percent Reduction	99.99%	99.99%	99.99%

Note: Due to unusually low counts for initial contact time, the inoculum concentration was used for calculation of percent reduction per client request.

Karen Barfoot

Departmental Review

Karen Alexander

Technical Review

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To Page No. 74Work done by *ViroMed*

Date

Witnessed

Recorded by *Roy S. Ding*

Date

Witnessed

PROPERTY OF KIMBERLY-CLARK CORPORATION

TITLE *Poly-UU Type-2*PROJECT NO. 57596
BOOK NO. P-7952

From Page No.

73 *Cuts**Ran***ViroMed** laboratories, Inc.ViroMed's biosafety laboratories
are becomingThis report is confidential. No part may be used for
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tested.Report Number:
G0823028Kimberly-Clark
1400 Holcomb Bridge Road
Roswell, GA 30076-2199

P.O. #: 15VB8653CB

Attn: Dr. Roger B. Quincy

GENERAL MICROBIOLOGY TEST REPORT

Sample Information: Fabric Sample, 7952-60-PB

Date Received:

Date In Test:

Date Completed:

Test Information:

AATCC Method 100 (modified)

Procedure #: GM210KMC.01

Culture Medium: Tryptic Soy Agar

Neutralizer Solution: Letheen Broth

Test Sample and Organism	<i>S. aureus</i> ATCC 6538	<i>E. coli</i> ATCC 8739	<i>P. mirabilis</i> ATCC 4630
Inoculum Concentration	8.4×10^6	4.7×10^7	3.4×10^6
Initial Contact Time	1.0×10^7	2.7×10^7	4.4×10^6
4 Hour Contact Time	5.0×10^1	3.0×10^1	2.5×10^1
Percent Reduction	99.99%	99.99%	99.99%

Karen Alexander

Departmental Review

Karen Alexander

Technical Review

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To Page No. 75

Work done by

ViroMed

Date

Witnessed

Date

Recorded by

Roger B. Quincy

Date

Witnessed

Date

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Poly1-Cl Type-2

74

*Cart*PROJECT NO. 57596
BOOK NO. P-7952*RBB*ViroMed's biosafety laboratories
are becoming
 Kimberly-Clark
 1400 Holcomb Bridge Road
 Roswell, GA 30076-2199

P.O. #: 15VB8653CB

Attn: Dr. Roger B. Quincy

GENERAL MICROBIOLOGY TEST REPORT

Sample Information: Fabric Sample, 7952-60-PA

Date Received:

Date in Test:

Date Completed:

Test Information:

 AATCC Method 100 (modified)
 Procedure #: GM210KMC.01
 Culture Medium: Tryptic Soy Agar
 Neutralizer Solution: Letheen Broth

Test Sample and Organism	<i>S. aureus</i> ATCC 6538	<i>E. coli</i> ATCC 8739	<i>P. mirabilis</i> ATCC 4630
Inoculum Concentration	8.4×10^4	4.7×10^7	3.4×10^6
Initial Contact Time	1.0×10^7	2.4×10^7	1.6×10^5
4 Hour Contact Time	$< 1.0 \times 10^1$	3.3×10^1	$< 1.0 \times 10^1$
Percent Reduction	99.99%	99.99%	99.99%

Katrina Tolok
Departmental Review*Karen Alexander*
Technical Review

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*ACT*To Page No. 76*Vinored*

Date

Witnessed

Date

Roger B. Quincy

Date

Witnessed

Date

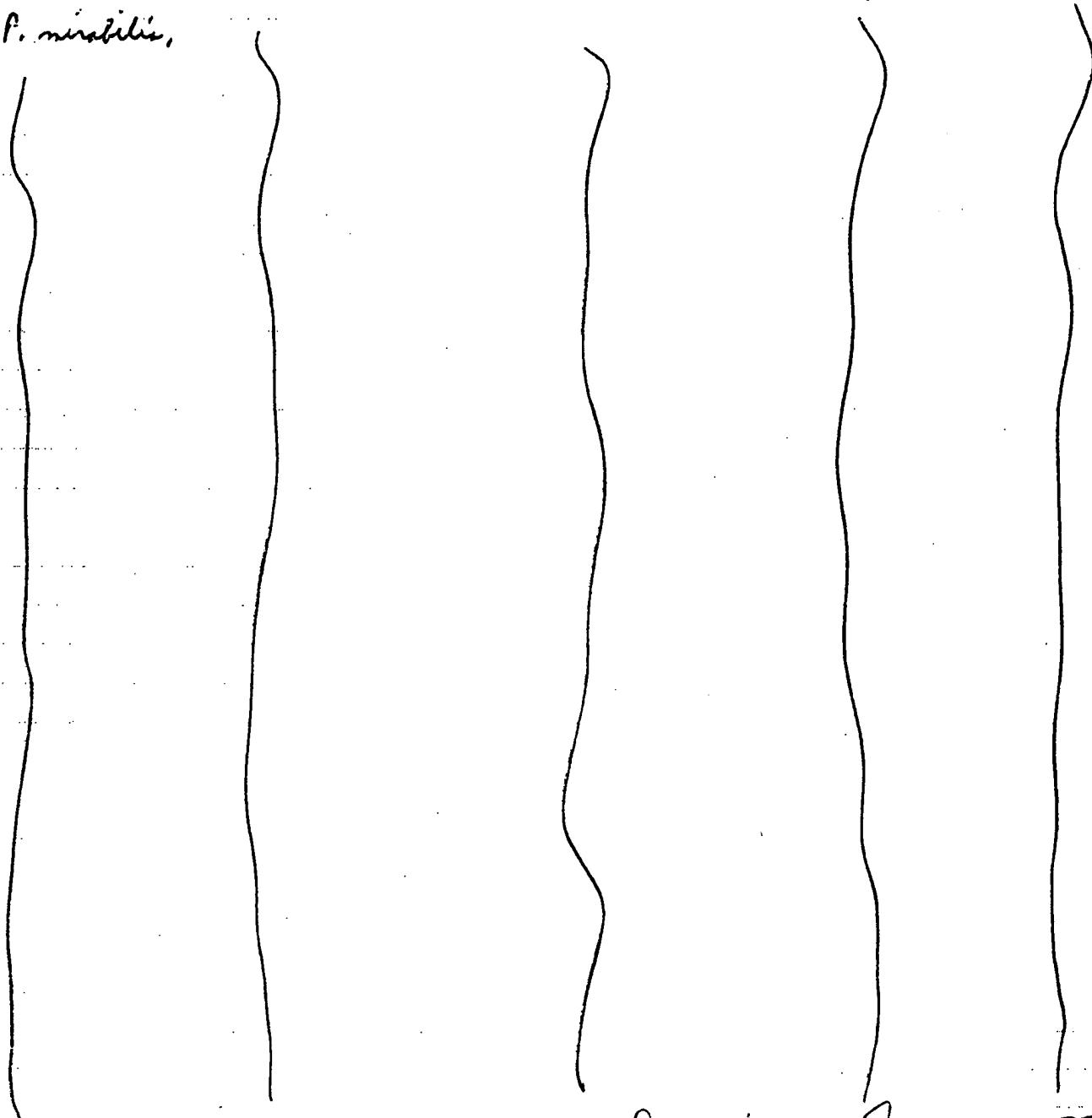
Proprietary. To Be Maintained in Confidence

57596
P-7952

Poly-Cl Type-2

75 Summary of Vibronic Results:

All of the 5% Poly-Cl Type-2 / fluff samples (i.e., 7952-60-PA, PB, PC and -PD) show excellent antimicrobial properties towards *S. aureus*, *E. coli*, and *P. mirabilis*.



Aug 22 1975
Aug 22 1975

77

title Poly-Cl Type-2

PROJECT NO. 57596

BOOK NO. A-7952

From Page No.

From Page No. 76 Drag Table Experiments to evaluate the behaviour about various
polymer samples for chlorine gas.

Cl Dosegr. Tube Expts. for Poly-Cl samples

180

From Page No. 76 Drag Tube Experiments to evaluate the dependence about various
Poly-Cl samples for chlorine gases. R8Q

Cl Drag Tube Expts. for Poly-Cl Samples

<u>Sample #</u>	<u>Description</u>
1A	"high Cl" Poly-Cl tested by Elih. Ben (AT) and J. Brooks (AC) in 0.0509 g seeded in 20cc headspace vial at 5:06 p.m. [REDACTED]
2B	Poly-1-Cl w/ 6inch, rec. 3/27/01 from J. Williams (Hobson) [REDACTED] 0.0499 g seeded in 20cc headspace vial at 5:11 p.m. [REDACTED]
3C	Poly-1-Cl type bldy. (HS-020-19-1), rec. from J. Williams (100g) on [REDACTED] 0.0500g seeded in 20cc headspace vial at 5:11 p.m. [REDACTED]

Diagram of set-up

20cc headspace vial
Teflon flexible plate
tube (1/16" ID, 5/16" OD, 1/16" wall)
middle ringed by orange hose fitting
deflected port ~2" \rightarrow
non-venting option position
drag tube for Cl 0.2-1 ppm
middle ringed by orange hose fitting
(1/8" to 1/16")
flexible plastic
orange spray nozzles

(continued on next page) [REDACTED]

Work done by R. Quigley, R. Brooks

Date of issue _____

Witnessed

Recorded by

Roger & Daing

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To Page No. 30

PROJECT NO. 57596

BOOK NO. P-7952

TITLE Poly-Cl - Type-2

From Page No. 77 Contd

RGA [REDACTED]

Cl Dross Test Expr. for Poly-Cl samples

Sample# time 60 ml volumes comments

1A 2:07p 1 tan-brown color developed in tube to reading of 0.2 ppm
 Syringe stays at 60 ml when let go (\Rightarrow no vacuum created)
 2:13p 1 more tan-brown color to \sim 1/3 between 0.2 and 0.5 ppm (\sim 0.3)
 2:16p 1 more tan-brown color almost reached 0.5 ppm (\sim 0.4)
 2:18p 1 more " " " at 0.5 ppm (\sim 0.4)
 2:19p 1 more " " " between 0.5 ppm and 1 ppm (cell lit 0.6 ppm) (\sim 0.7 ppm)

Note: Cut new piece of tygon tubing before sample needle and new drosser tube before analyzing sample 2B.

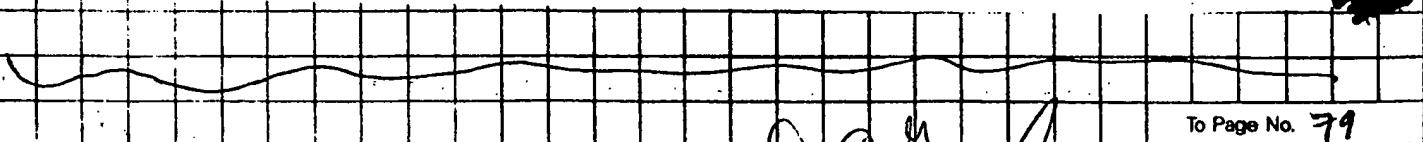
2B 2:31p 1 tan-brown color developed rapidly and was above the 3 ppm MAX. reading after completing 60 ml stroke; syringe stays at 60 ml when let go (\Rightarrow no vacuum created).

Note: Sample "1A" was lighter in color (more tan, more white) than sample "2B" or "3C".

3C Is this due to more Cl in samples "2B" and "3C"?

3C 2:50p 1 tan-brown color developed rapidly and was above the 3 ppm MAX. reading after completing the 60 ml stroke; syringe stays at 60 ml when let go (\Rightarrow no vacuum created).

Note: Next test Vm 6el 0 w/ Poly-Cl for removal of residual chlorine.

				To Page No. 79
Work done by	Roy B. Dury	Date	[REDACTED]	Witnessed
Recorded by	Roy B. Dury	Date	[REDACTED]	Witnessed
Proprietary. To Be Maintained in Confidence				

TITLE Poly-Cl Type-2

PROJECT NO. 57596
BOOK NO. P-7952

From Page No. 78 (Contd)

████████ → Cl Dose Tube Expt for new Poly-Cl (i.e. Poly-Cl Type-2) R&D █████

- using Poly-Cl Type-2 (HS039-77-3, 54.3 g) → received from Heubelmark

in █████

Sample #

080301-1 → 0.0521 g plated in 20cc beaker vial at 11:11A █████

At ~4:18p █████ tested for Cl as follows: (Note: using same setup as described for █████ Expt.

time	blank volume	Comments	See beaker vial (no book)
------	--------------	----------	---------------------------

4:42p	1	no color	
4:43p	1 more	no color	
4:48p	1 more	" "	
4:50p	1 more	" "	
4:52p	1 more	single a.v. at time of the yellow color had develo loped to a new time. The color was 0.1448 well below first mark of 0.2 ppm	

080301-5 → 0.0501 g Poly-Cl type 2 + 0.0485 g saline plated in 20cc

beaker vial at 11:18A █████ Note → This amount of saline
resulted in a small amount of liquid expanding about 1/10 of the beaker in
the case of the vials, the other 9/10 of the beaker expand to stick to
the bottom of the vial, but liquid was not obvious. Did the poly-Cl
absorb/absorb some of the saline?

time	blank volume	Comments
5:00p	1	* all yellow color has developed to before the first
5:04p	1 more →	the all yellow color is diffuse but clear 0.1 ppm
5:06p	1 more →	1 way to the first mark (0.2 ppm),
5:12p	1 more →	diffuse but almost up to the 0.2 ppm mark
5:13p	1 more →	" " " " " " "

Note: The beakers in this vial are light yellow in color compared to the white beakers in sample 080301-1.
Lighter liquid is lighter, * need to save enough Cl until we need for sample 080301-1.
free at the edge of the beaker (Contd next page)

To Page No. 80

Work done by Roger B. Doring

Date █████ Witnessed █████

Recorded by

Roger B. Doring

Date █████ Witnessed █████

Date █████

PROPERTY OF KIMBERLY-CLARK CORPORATION

PROJECT NO. 57596
BOOK NO. P-7952TITLE Poly-Cl Type-2From Page No. 79cont.RBCCont. Cl Degr. Tube Expts. for Poly-Cl Type-2

The rest of sample + sample that was too connected to the drug tube assembly (already one removed) was left inserted into the headspace vial for 081601-S to see if any corrosion occurs (as seen for sample 3C for [REDACTED] Cl Degr. Tube Expts. → see Headspace-GL-2000 book).

8/16/01 More Cl Degr. Tube Expts. for Poly-Cl Type-2Sample #

081601-1 → 0.0505 g. sealed in 20cc headspace vial at 4:31p [REDACTED]

At 4:31p, put 081601-1 into GL#1 oven w/ lid closed at 50°C.

At 4:32p, removed the drug tube assembly (see diagram in Headspace GL-2000 book lid [REDACTED])

and pulled to form 60 ml volume from the drug tube. The tube became only v. slightly yellow-colored about $\frac{1}{2}$ after the first minute + the first reading of 0.2 ppm. The color was not continuous; it was very diffuse (i.e. the yellow color was dispersed amongst uncolored (white) matrix (particles) in the tube). The Poly-Cl Type-2 particles are the same color (white to off tan/white) as the original particles after being heated to 50°C and cooled to room temp. RBC

Conclusion | The new Poly-Cl (i.e. Poly-Cl Type-2) is considerably more stable in terms of releasing chlorine gas than the previous version (see pp. 77-78, P-7952). Need to work on potential instability when the Poly-Cl Type-2 comes in contact with liquid (see above results on p. 79, P-7952).

To Page No. 119Work done by Rough B. Doring, R. B. Doring

Witnessed

Recorded by Rough B. Doring

Date

Witnessed

Date

TITLE Poly-Cl Type-2

PROJECT NO. 57596
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From Page No.

80

More hydrogen chloride data for Poly-Cl Type-2:

~~_____~~ → observation for 080301-S

RBC

080301-S had two needles submerged in the septa into the hydrogen rich pipe
~~_____~~ why these needles were removed and observed for effects of corrosion (see facing
page) ~~_____~~ AET

~~_____~~ → observation for 080301-S

RBC

Both needles showed small spots brown in color that coincided with the
part of the needle that was in the septa material. There was not
any discoloration (indicative of corrosion from hydrogen Cl₂) on the
part of the needles that was submerged inside the hydrogen rich (with
the alum and Poly-Cl Type 2 particles).

This result is different from the previous Poly-Cl (type 1), in which
a large amount of corrosion was observed for the submerged needle in

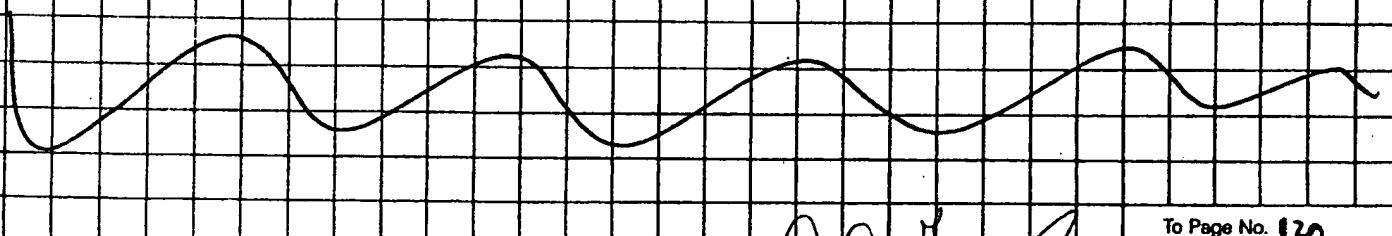
The hydrogen rich with the Poly-Cl type 1 particles. Note → the
Poly-Cl type 1 results showed lots of Cl₂ in the hydrogen

(see Cl data results for ~~_____~~),

in the Hydrogen bc book.

AET

Note: See p. 79, P-7952 for the hydrogen chloride results in the 080301-S sample.



To Page No. 120

Work done by Roger B. Davis

Date ~~_____~~Witnessed ~~_____~~

Recorded by Roger B. Davis

Date ~~_____~~Witnessed ~~_____~~

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TITLE Poly-Cl Type-2

PROJECT NO. 57596
BOOK NO. P-7952

From Page No. 119

Contd

R82

████████ → Cl Dose, Tube Expts for Poly-Cl Type 2

- using Poly-Cl Type-2 (HS079-77-3, 543g) → received from Heiblum a

Sample #

101801-1 → 0.0504 g Poly-Cl Type 2 + 0.1455 g saline pulled in 2cc syringe
vial at 3:58p █████

At 3:58p, put 101801-1 into GC+1 oven w/ lid closed at 37°C.

At 4:12p, Committed the drug tube assembly (see diagram in Heiblum-GC 2000 book dated █████)
and pulled ^{used new tubing, 2 needles to vial were new, and no drug/tube} fine 60 ml volume thru the new drug tube. Comparison to a fresh tube
against a white background showed a v. slight trace (slight yellowish color
"differ") next to the first mark, between the first mark and the first reading of 0.2 ml.
This should really be considered non-detectable.101801-2 → 0.0500 g Poly-Cl Type 2 + 0.1462 g saline pulled in 2cc syringe
vial at 4:03p █████████ → At 4:33p, put 101801-2 into GC+1 oven w/ lid closed and temp heating oven from
37°C to 50°C, oven was at 50°C at 4:34p (actually a readout to 57°C, back to
50°C when checked at 4:40p)Note: Used ^{new} same drug tube for this sample, but used the same needles
as new ^{+ tubing} and for sample 101801-1.At 4:51L 4:55p, pulled ^{new} fine 60 ml volume thru the committed new drug tube, on
Comparison to a fresh tube against a white background again showed ^{new} trace
v. v. sl. trace of the yellow color just like described for tube 101801-1.

To Page No. 121

Work done by Roger B. Dwyer

Date █████

Witnessed

Date █████

Recorded by Roger B. Dwyer

Date █████

Witnessed

Date █████

PROPERTY OF KIMBERLY-CLARK CORPORATION

PROJECT NO. 57596

BOOK NO. P-7952

TITLE Poly1-Cl Type-2

From Page No. 120

Summary of all Headspace-Chlorine Data for various Poly1-Cl samples:

RBR

Quincy, Roger

From: Quincy, Roger
 Sent: Tuesday, [REDACTED] 11:25 AM
 To: Laird, Sarah; Evans, Eric; Hansen, Dick; Berrier, Phillip; Wyatt, Nancy
 Cc: Gadsby, Elizabeth; Edens, Ron; Pike, Dan; Everhart, Cherie; Zabronsky, Jerry
 Subject: Poly1-Cl Headspace Chlorine Data

Here are the headspace chlorine data for the various Poly1-Cl samples, including the "wet, elevated temp." samples (requested from Product Safety Review Meeting). Please let me know if there is any other testing required for our requested safety clearance (for urine odor panel study)?

Poly1-Cl Type-2
Headspace Cl ...Headspace Chlorine Results for Poly1-ClBackground:

Drager tube CH 24301 (Chlorine 0.2/a) with a standard measuring range of 0.2 to 3 ppm was used to determine headspace chlorine levels for various Poly1-Cl samples. In the presence of chlorine gas (Cl₂), the tube will undergo a color change from white to yellow-orange. Bromine, chlorine dioxide, and nitrogen dioxide will also cause this color change. For an actual chlorine concentration of 0.2 to 3 ppm to be read from the tube, the requirement is for 10 strokes to be pulled through the tube using a Drager bellows type pump. Each stroke supplies 100 cm³. If only one stroke or 100 cm³ is pulled through the tube, then the measuring range for the tube will be 2 to 30 ppm. For the various Poly1-Cl samples, Poly1-Cl powder alone and in combination with saline was placed in a 20-cm³ headspace vial. The vial was crimped shut and left at ambient temperature for a desired length of time. Then, the vial was either tested for headspace chlorine or first placed in a GC oven at elevated temperature for a desired length of time before being tested. The headspace from the vial was tested for chlorine by piercing the septum of the vial cap with a needle that was attached to the Drager tube with rubber tubing. The headspace was removed with a 60-cm³ syringe that was attached to the other end of the Drager tube with rubber tubing. The septum of the vial cap was also pierced with a second needle in order for ambient air to replace the removed headspace air above the sample. The amount of headspace chlorine for a sample was calculated from the Drager tube reading, the number of 60-cm³ volumes removed, and the relationship that 1000 cm³ (10 strokes) must be pulled through the tube in order to read chlorine in a range of 0.2 to 3 ppm.

RBR

[REDACTED]

ACG

To Page No. 122

Work done by

Roger B. Quincy

Date

Witnessed

Recorded by

Roger B. Quincy

Date

Witnessed

Date

TITLE Poly1-Cl Type-2

PROJECT NO. 57596
BOOK NO. P-7952

From Page No. 121

Cont. *Summary*

RBO

Results:

The following table shows the headspace chlorine data for various Poly1-Cl samples.

Sample	Details	Tube Reading	Calculated Amount	Calculated per Piece ¹
Poly1-Cl Type-1, Dry	12 days at room temp.	> 3 ppm	> 50 ppm	> 68 ppm
Poly1-Cl Type-2, Dry	6 days at room temp.	<<< 0.2 ppm	<<< 0.7 ppm	<<< 0.9 ppm
Poly1-Cl Type-2, Wet	+ 300% saline, 6 days at room temp.	< 0.2 ppm ²	< 0.7 ppm ²	< 0.9 ppm ²
Poly1-Cl Type-2, Dry, Elevated Temp.	1 day at room temp., 20 min. at 50°C	<< 0.2 ppm	<< 0.7 ppm	<< 0.9 ppm
Poly1-Cl Type-2, Wet, Elevated Temp.	+ 300% saline, 1 day at room temp., 30 min. at 37°C	<<< 0.2 ppm	<<< 0.7 ppm	<<< 0.9 ppm
Poly1-Cl Type-2, Wet, Elevated Temp.	+ 300% saline, 1 day at room temp., 20 min. at 50°C	<<< 0.2 ppm	<<< 0.7 ppm	<<< 0.9 ppm

¹ "Calculated per Piece" was determined by taking the "Calculated Amount" of chlorine, dividing by the weight of Poly1-Cl in the headspace tube (ca. 0.05 g), and then multiplying by the amount of Poly1-Cl in a 3-inch diameter piece of 600 gsm fluff that contains 2.5 wt% Poly1-Cl (0.0684 g). The urine odor panel study will use 3-inch diameter pieces of materials.

² These values were determined from a Drager tube that had been previously used for the "Poly1-Cl Type-2, Dry" sample. Therefore, the reported values are probably higher than values that would have occurred with a new tube. New tubes were used for all other samples.

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Work done by	Rough & Dailey	Date	Witnessed	Date
Recorded by	Rough & Dailey	Date	Witnessed	Date